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THE EFFECTS OF MINOR CONSTITUENTS IN CALCIUM SILICATE  
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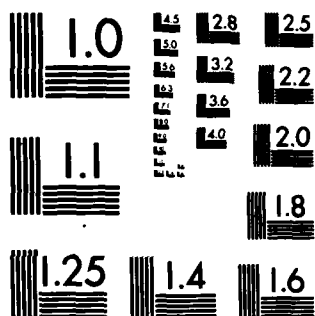
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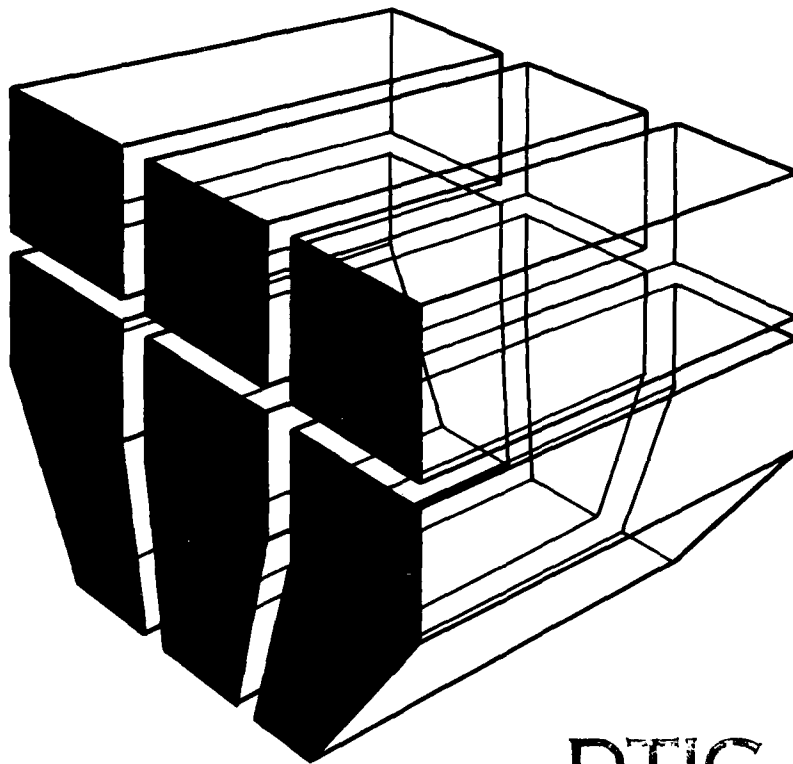


Technical Report M-346  
June 1984

**THE EFFECTS OF MINOR CONSTITUENTS IN CALCIUM SILICATE  
INSULATION ON THE CORROSION OF UNDERGROUND  
HEAT DISTRIBUTION SYSTEMS**

**AD-A143 378**

by  
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1. Prevent water from entering the system through manholes by providing and maintaining effective seals at manhole entries.
2. Insure positive gravity drainage of the system by maintaining positive slope to prevent water from accumulating at low spots.
3. Minimize contamination of the system by keeping soil and debris out of the conduit during installation.
4. Periodically check systems for high water in manholes, evidence of water in the conduit, and evidence of corrosion.
5. Promptly drain conduits if flooding occurs.
6. Promptly repair leaks in the conduit or carrier pipes to prevent further contamination of the system from ions in the environment.

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## FOREWORD

This study was conducted for the Directorate of Engineering and Construction, Office of the Chief of Engineers (OCE), under Project AT45, "Energy and Energy Conservation"; Technical Area B, "Installation Energy Conservation Strategy"; Work Unit 011, "Modernization of Underground Heat Distribution Systems." The OCE Technical Monitor was Mr. Dale Otterness, DAEN-ECE-E.

This research was performed by the Engineering and Materials (EM) Division of the U.S. Army Construction Engineering Research Laboratory (CERL). Dr. R. Quattrone is Chief of CERL-EM.

COL Paul J. Theuer is Commander and Director of CERL, and Dr. L. R. Shaffer is Technical Director.

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# THE EFFECTS OF MINOR CONSTITUENTS IN CALCIUM SILICATE INSULATION ON THE CORROSION OF UNDERGROUND HEAT DISTRIBUTION SYSTEMS

## 1 INTRODUCTION

### Background

Asbestos-free calcium silicate insulation has been used almost exclusively for prefabricated steel conduit underground heat distribution systems since the mid-1970s, when safety problems prevented the use of asbestos-containing calcium silicate insulation. Calcium silicate insulation was selected because of its chemical inertness and its ability to withstand the effects of boiling water for long periods of time without mechanical damage. However, some users believe that this type of insulation has caused pitting corrosion of heat distribution systems. Although this problem does not appear to be widespread, its consequences can lead to serious difficulties in the future, when many systems will contain calcium silicate insulation.

### Objectives

The objectives of this study were (1) to assess the detrimental effects of minor constituents in calcium silicate insulation on the corrosion of prefabricated steel conduit heat distribution systems, and (2) to recommend ways to minimize this type of corrosion.

### Approach

Samples of pitted prefabricated steel conduit heat distribution systems were analyzed. Samples of as-received calcium silicate insulation were obtained from three major manufacturers for chemical analysis; the leachable chloride content of each sample was determined in the laboratory after prolonged boiling tests. The effects of the observed leachable chloride concentrations in the insulation materials on the corrosion of heat distribution systems were determined. The factors contributing to the corrosion of underground heat distribution systems were reviewed, and recommendations for minimizing the effects of corrosive species on the systems were made.

### Mode of Technology Transfer

This study will impact the revision of Corps of Engineers Guide Specification CEGS 15705, *Underground Heat Distribution Systems*; Naval Facilities Engineering Guide Specification NFGS 15705, *Underground Heat Distribution Systems*; and Air Force

Pamphlet 88-44, *Underground Heat Distribution Systems*.

## 2 THE EFFECT OF LEACHABLE IONS IN CALCIUM SILICATE INSULATION ON PITTING CORROSION

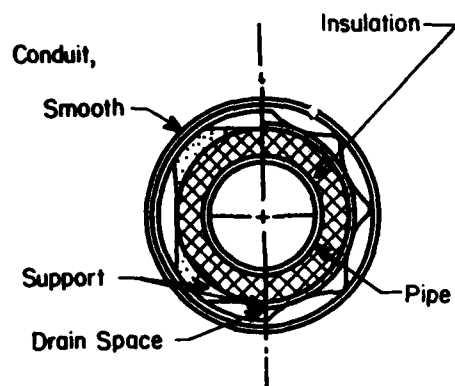
The presence of leachable ions in materials used to insulate metals can cause various types of corrosion. Serious corrosion of metals can occur when they are exposed to moisture and elevated temperatures.

Figure 1 shows a typical prefabricated steel conduit heat distribution system. A carrier pipe is placed in a larger conduit pipe, and insulation is put between the pipes. Military construction specifications require drainable and dryable systems which in most cases results in a 1-in. (25.4 mm) air space between the insulation and the conduit.<sup>1</sup> At the entry of the system into a manhole, an endplate is welded to the conduit as a seal against water; the carrier pipe continues into the manhole, and vent and drain ports are provided for the conduit at the system entry.

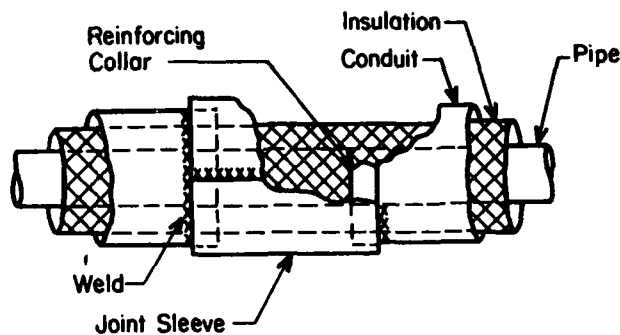
Before about 1975, asbestos-based calcium silicate insulation was used for underground heat distribution systems. However, safety problems associated with the use of this material led to the development of the calcium silicate insulation now used. Calcium silicate was selected because of its chemical inertness and because it maintains its physical integrity during boiling, which can occur if the conduit floods.<sup>2</sup>

<sup>1</sup> *Military Tri-Services Specifications for Underground Heat Distribution Systems*: Army and Navy documents CE-301.21, TS-P28g, respectively, and Air Force Pamphlet No. 88-007-1.

<sup>2</sup> *Standard Specification for Calcium Silicate Block and Pipe Thermal Insulation*, ANSI/ASTM C 533-72 (American Society for Testing and Materials, 1972); B. H. Van Dyke, "Considerations for Design and Selection of Low Temperature Piping Insulation Systems," Paper 76-T-43, *Proceedings* (American Gas Association, Operating Section, Arlington, VA, May 3-5, 1976), p. 13; H. Oppermann, "Resistance to Chemical Action by Calcium Silicate Bricks," *Autoclaved Calcium Silicate Building Products*; papers read at the symposium (1967), pp. 155-62; V. Kahol, "Calcium Silicate Insulation," *Encyclopedia of Chemical Processing and Design*, Vol. 6 (1978), pp. 64-72; H. V. Shah and D. J. Mehta, "Preparation of Calcium Silicate Moulded Insulation," *Research and Industry*, Vol. 14 (April-June 1969), pp. 62-5; K. D. Dhariyal and M. Rai, "Calcium Silicate Bonded Asbestos Insulation Products," *Research and Industry*, Vol. 24 (June 1979), pp. 90-101.



TYPICAL CROSS SECTION



TYPICAL FIELD JOINT

Figure 1. Prefabricated steel conduit system.

However, some users feel that calcium silicate insulation is contributing to the corrosion of conduit shells. It has been suggested that ions which leach from the insulation are responsible for the corrosion. Where problems were observed, severe pitting of conduit shells was noted, but little or no general corrosion was evident. Pitting corrosion of underground heat distribution systems is a potentially serious problem because leaks may result in large losses of thermal energy, and the system may eventually become unusable.

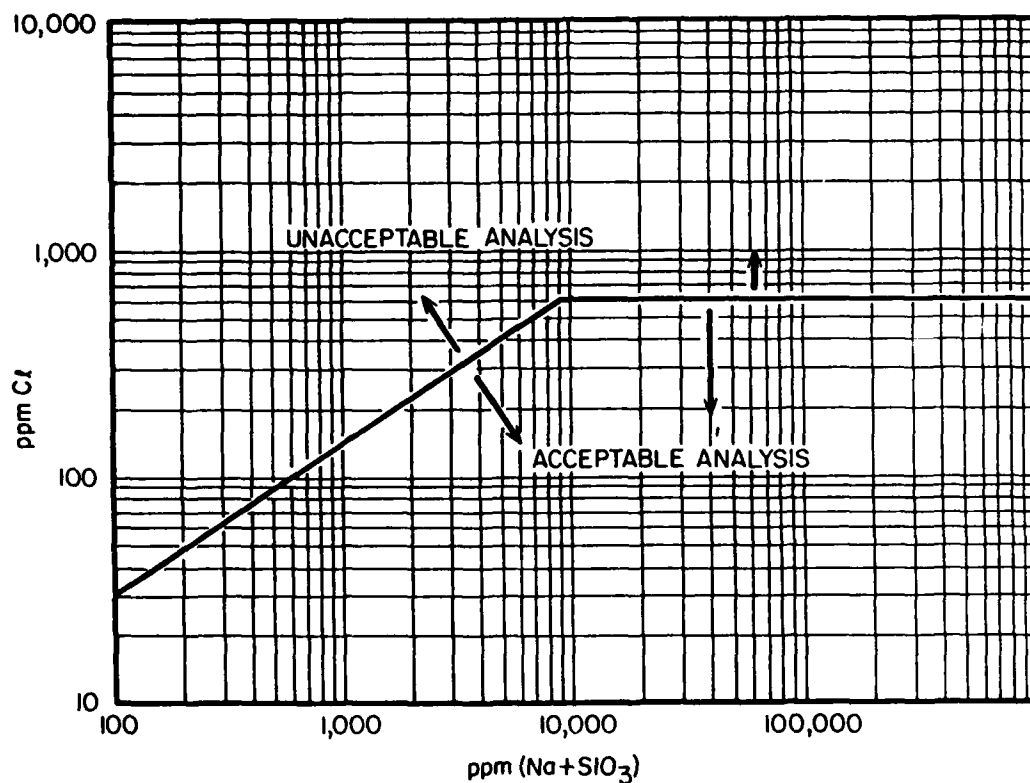
Although various mechanisms have been proposed to explain the problems observed, this type of corrosion is probably the result of pitting by halide ions. It is known that pitting corrosion of steel is caused by chlorides and, to a lesser extent, by other halide ions such as fluorides.<sup>3</sup> Other ions, such as magnesium,

usually cause general corrosion with little or no pitting, because the resistivity of electrolytes in contact with metals is reduced.

Pitting corrosion of metals can be controlled by minimizing chloride and other halide ion concentrations, reducing the amount of free oxygen, adding alkalis to chloride-containing environments, applying cathodic protection, and/or operating the systems at the minimum required temperatures. Although these techniques are effective in reducing the pitting of many metals, the configuration of underground heat distribution systems requires reducing the amount of chlorides. If conduit flooding occurs, chlorides and other detrimental ions can enter the system with groundwater or contaminated water from a manhole, or the ions can be leached from the insulation. There is little control over the composition of water that enters a conduit from groundwater or a manhole; however, the amount of leachable components in the insulation can be controlled during processing.

The detrimental effect of chlorides from materials used to insulate metals is well known, and there are standard techniques for evaluating their corrosivity.

<sup>3</sup>H. G. Fontana and N. D. Greene, *Corrosion Engineering*, B. J. Clark and F. A. Neal, eds. (McGraw-Hill Book Co., 1978), Chapters 3 and 5; H. H. Uhlig, *Corrosion and Corrosion Control: An Introduction to Corrosion Science and Engineering* (John Wiley and Sons, Inc., 1967), Chapters 6, 17, and 18.



**Figure 2.** Acceptability of insulation materials on basis of chloride, sodium, and silicate contents.

Military Specification MIL-I-24244A<sup>4</sup> gives ways to analyze calcium silicate insulation for leachable chlorides, sodium, and silicate. Acceptance of the material depends on both the chloride content and the combined sodium and silicate content, because the sodium and silicate mitigate the adverse effects of the

chlorides. Figure 2 shows the leachable-ion limits established by MIL-I-24244A for insulation, the limits of chloride solubility, and the mitigating effects of sodium and silicate.

In the standard procedure, 20-g specimens that are representative of the sample cross-section are pulverized in a Waring blender at high speed for 30 to 60 sec. They are then boiled for 1/2 hour, cooled, filtered, and diluted with wash water to 500 mL. The filtrate is analyzed for chloride, sodium, and silicate ions.

<sup>4</sup>Insulation Materials, Thermal, With Special Corrosion and Chloride Requirements, Military Specification MIL-I-24244A (Department of Defense, 12 February 1974).

### 3 PROCEDURE

#### Examination of Pitted Samples From the Field

Samples of pitted conduit pipe and adjacent insulation were obtained from Fort Bragg, NC, and Fort Gordon, GA, for observation. A commercial laboratory performed chemical analyses of the corrosion products in and around the pits as well as of material away from the pits.

#### Examination of As-Received Calcium Silicate Insulation

Samples of conventional calcium silicate insulation for underground heat distribution systems were obtained from Johns-Manville, Pabco, and Owens-Corning. The samples, which were randomly labeled A, B, and C, were chemically analyzed by a commercial laboratory.

#### Boiling Tests of Insulation To Determine Leachable Chlorides

A procedure similar to that described in Military Specification MIL-I-24244A, but of longer duration, was used to determine leachable chlorides in the insulation materials. Specimens for the investigation were cut from the interior of the bulk to avoid surface contamination. The specimens were powdered using a mortar and pestle.

Samples weighing between 10 and 40 g were weighed to the nearest 0.001 g and added to 400 to 900 mL of deionized-distilled water. The suspensions of insulation in water were boiled in flasks over a hot plate while being stirred with a magnetic stirrer for 2 to 200 hours. The level of boiling liquid was maintained by either adding more distilled-deionized water to the suspension, or by using condensing coils placed over the flasks. Distilled-deionized water samples containing no calcium-silicate insulation were run simultaneously as control tests.

After the suspensions were boiled for the desired time, they were cooled and filtered with a Buchner vacuum funnel to remove the suspended calcium silicate powders. The filtrate volume was reduced to 5 to 20 mL by boiling to concentrate the chlorides, and the final volume was measured to the nearest 0.1 mL.

The milliequivalent of chloride ions per liter\* in the filtrate was determined using a Corning Model 920M Chloride Meter. At least ten readings were made for each sample. Samples were diluted, and the process was repeated until the chloride concentration was too small to measure. The amount of chloride leached from each sample was determined by calculating the total chlorides dissolved during boiling. The leachable chloride content per unit weight of insulation was determined as the ratio of total chlorides in the filtrate solution after boiling to the initial weight of the insulation powders.

### 4 RESULTS AND DISCUSSION

#### Chemical Analysis of Pitted Conduit Pipes

Table 1 describes some of the samples of pitted conduit pipe and associated insulation obtained from Fort Bragg, NC, and Fort Gordon, GA. Table 2 shows the results of chemical analysis of these samples. Large quantities of chlorides but only minimal amounts of fluorides and other halogen ions were observed near the corrosion pits. This suggests that chlorides were the major species contributing to pitting.

#### Chemical Analysis of As-Received Calcium Silicate Insulation

A commercial laboratory chemically analyzed as-received samples of Johns-Manville, Owens-Corning,

\*Milligrams/liter equals parts per million by weight, assuming that the density of water is 1 gram/cubic centimeter.

Table 1  
Description of Pitted Samples of Pipe

Sample No.	Source	Sample Description
4	Fort Gordon	Corrosion products near pitted conduit
5	Fort Bragg	Bulk insulation near pitted conduit
6	Fort Bragg	Bulk interior of calcium silicate insulation

**Table 2**  
**Chemical Analysis of Pitted Samples**  
**From Fort Bragg, NC, and Fort Gordon, GA**

	Sample Number		
	4	5	6
Chloride*	192.5/192.5	184.0/160.0	155.0/157.5
Fluoride*	20.5/16.0	32.5/20.5	19.5/20.5
Sodium*	1180/1215	520/500	520/500
Magnesium*	<10/<10	<10/<10	<10/<10
Silicate*	6339/6231	3034/2709	3034/2709

\*Concentration in micrograms per gram, or ppm by weight.

and Pabco preformed calcium silicate insulation. The samples were randomly labeled A, B, and C.

Table 3 shows the chemical analysis results for the three types of commercially available calcium silicate insulation. The chloride concentrations were found to range from 217 to 340  $\mu$ g chloride per gram insulation (i.e., ppm chlorides by weight). Insulation A and Insulation B contained fewer than 5 ppm fluorides, while Insulation C contained fewer than 100 ppm fluorides. Insulations A, B, and C were found to contain an average of 51, 190, and 6200 ppm of sodium, respectively. The high levels of sodium in Insulation C may mitigate the effects of its leachable chloride ions on pitting corrosion; however, the sodium ions may also contribute to general corrosion by lowering electrolyte resistivity.

These results suggest that pitting corrosion of underground heat distribution systems by fluoride ions is a secondary effect of pitting by chloride ions. Not all of the chlorides and fluorides in the calcium silicate insulation are soluble in boiling water, since these ions may become covalently bonded and trapped within the calcium silicate structure. These types of bonded cations are released only in concentrated acid solutions or at

high temperatures (i.e., temperatures above one-half of the melting point in degrees kelvin, or about 800°C for calcium silicate).

#### **Determining the Concentration of Leachable Chlorides**

Table 4 shows the results of the tests conducted to determine the solubility of chlorides in calcium silicate insulation during prolonged boiling. Table 5 summarizes the results by manufacturer. Chloride concentrations of the filtrate were below detectable limits for boiling times of 1 to 2 hours; however, after 7 or 8 hours, the chloride concentrations of the filtrate increased greatly. Increasing boiling time from 7 to 175 to 200 hours did not appreciably increase the chloride concentration. There was appreciable scatter in the data, which was attributed to the lack of equipment sensitivity at the low chloride concentrations being measured. Control tests in which distilled-deionized water samples containing no insulation powders were boiled for up to 175 hours were conducted; the results showed that chloride concentrations remained below the detectable limit. These results suggest that significant amounts of soluble chlorides are released during prolonged boiling of calcium silicate insulation. The average values of soluble chloride concentrations in the

**Table 3**  
**Chemical Analysis of As-Received Calcium Silicate Insulation**

Manufacturer	Chlorides*	Fluorides*	Silicates*	Sodium*
A	217.5/237.5	4.73/4.28	9700/10,300	51/50
B	349/315	3.69/2.86	12,800/10,000	190/190
C	247.5/255	64.3/93.5	7600/8400	6300/6100

\*Concentration in micrograms per gram, or ppm by weight.

**Table 4**  
**Results of Boiling Tests of Calcium Silicate Insulation**  
**to Determine Leachable Chloride Concentration**

Sample	Weight (grams)	Manufacturer	Boiling Time (hours)	Milligrams Chloride Ions* Per Gram of Insulation
B	36.3951	C	7.75	53
C	42.3795	C	7.75	58
D	31.6708	A	7.5	66
F	32.2570	A	7.5	36
F	31.6161	B	9.5	196
G	38.6751	B	9.5	161
H	32.5617	B	7.5	72
I	27.3751	B	7.5	66
M	39.5298	B	27.5	73
N	30.7681	B	17.5	118
O	37.2564	B	23	111
P	16.2123	B	20	131
Q	16.6750	B	21	163
R	16.9609	B	10	68
S	17.8387	B	9	137
T	16.3329	B	16.2	128
U	16.8900	B	192	155
V	16.0417	B	172	71
W	16.7743	B	172	84
X	16.6655	B	172	111
Y	16.3424	C	187	54
Z	17.3658	C	193	51
AA	15.8158	A	187	41
BB	16.0858	A	187	45
CC	15.4201	A	189	29
DD	15.9280	C	191	117
EE	16.6596	A	191	18
FF	17.2426	C	173	36
GG	13.5068	B	41	230
HH	15.1350	B	41	176
II	15.9924	B	41	163

\*Milligrams of chloride/gram insulation = ppm by weight.

three insulation materials ranged from  $39.1 \pm 16.2$  to  $127 \pm 47.8$  ppm for boiling tests ranging from 7 to 200 hours.

#### Estimating Chloride Concentrations in a Flooded Conduit Pipe

The effect of the leachable chloride levels observed in the laboratory on the corrosion of underground heat distribution systems was assessed by estimating the chloride concentration of water in a conduit after flooding. It was assumed that if there is partial conduit flooding of the type that could be expected if a conduit or carrier pipe leak occurs, or if water from a manhole backs up into a conduit, continued boiling would dissolve all of the soluble chlorides within a few

days. This assumption was based on the severity of the environment near the boiling water. As boiling progresses, the water level in the conduit may rise or fall, depending on the rate of water leakage into the system

**Table 5**  
**Summary of Laboratory Tests**  
**for Leachable Chlorides**  
**in Commercial Calcium Silicate Insulation**

Manufacturer	Soluble Chlorides (ppm by weight)
A	$39.1 \pm 16.2$
B	$127.1 \pm 47.8$
C	$61.5 \pm 28.2$

and the rate of water evaporation during boiling. It should be noted that chlorides and other ions in solution become more concentrated during boiling, since they do not evaporate with the water.

Figure 3 is a cross-section of a flooded conduit, which shows that the volume of water in the conduit is

a geometrical function of the water depth. Figure 4, which shows this function graphically, assumes that the ratio of the interior radius of the conduit to the exterior radius of the carrier pipe is 2.33.

The chloride concentration of water in a conduit system as a function of water depth and of leachable

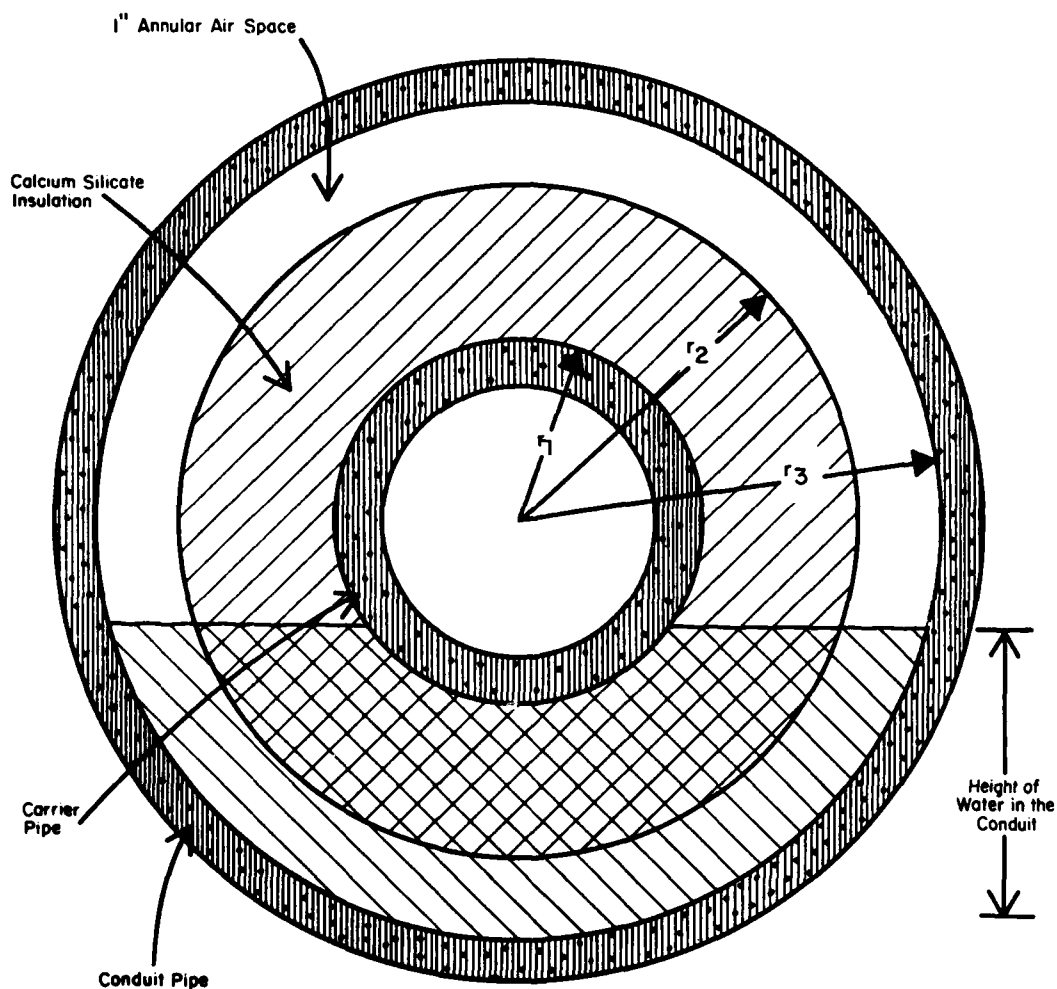
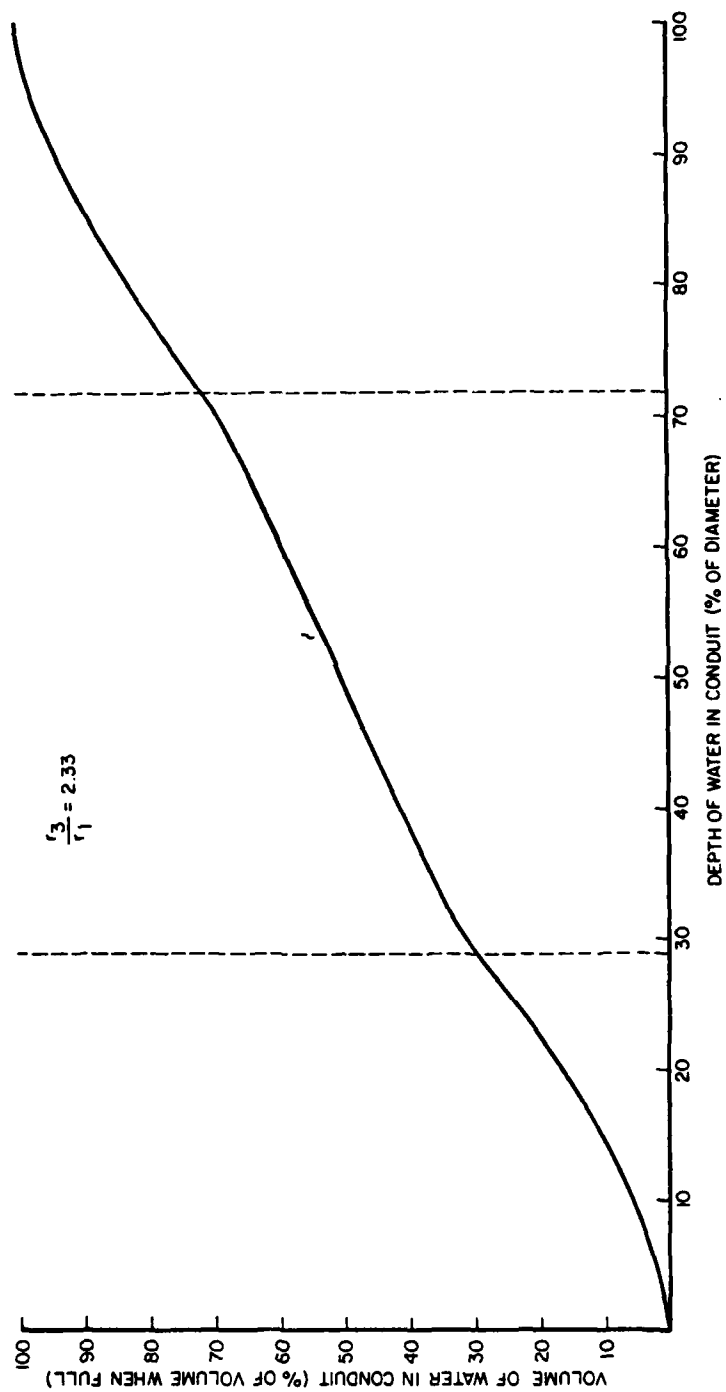


Figure 3. Cross-section of flooded conduit.



**Figure 4.** Effect of changes in height of water in a conduit on volume of water in the conduit.



chloride content of the insulation was estimated. The following was assumed:

1. The volume of the conduit occupied by the calcium silicate is negligible, since the insulation is about 85 percent porous.

2. The effect of system slope on the chloride concentration would be neglected.

Both of these assumptions led to a conservative estimate of chloride concentrations.

Figure 5 shows a plot of this function for a typical conduit. The example system for this calculation was a 4-in. (101.6-mm) nominal-diameter carrier line in a 10-in. (254-mm) nominal-diameter conduit pipe with 2 in. (50.8 mm) of insulation and a 1-in. (25.4-mm) annular air space. Table 6 lists the values for pipe diameters and leachable chloride levels used for the calculations.

The insulation materials tested in the laboratory had leachable chloride concentrations ranging from about 40 to 130 ppm chlorides. Figure 5 shows that for insulation containing 50 to 100 ppm leachable chlorides, the chloride concentration of water in a flooded conduit increases dramatically to more than 100 ppm when the water level in the conduit falls to less than 1 in. (25.4 mm). This suggests that after prolonged boiling, concentration of chlorides is a significant problem. During boiling, the decrease of water volume in the conduit can change the concentration of chlorides in the water by several orders of magnitude. Thus, it is evident that the concentration of chlorides in water flooding a conduit depends greatly on the volume of the water in the conduit, but is less dependent on the solubility of chlorides in the insulation material.

#### Techniques for Minimizing the Corrosion of Underground Heat Distribution Systems

The corrosion of underground heat distribution systems by aggressive ions from both the insulation and the environment can be mitigated by appropriate design, construction, and operation and maintenance techniques.<sup>5</sup>

During system design and construction, many long-term problems can be avoided by taking steps to mini-

<sup>5</sup>Ellen Segen and Ching Ping Chen, *Investigation of Tri-Service Heat Distribution Systems*, Technical Report M-347 (U.S. Army Construction Engineering Research Laboratory, 1984).

**Table 6**  
**Tabulation of Values Used to Determine the Concentration of Chlorides as a Function of the Depth of Water in a Flooded Conduit (Metric Conversion Factor: 1 in. = 25.4 mm)**

Nominal diameter of carrier pipe	4 in.
Thickness of insulation	2 in.
Nominal diameter of conduit pipe	10 in.
$r_1$ = exterior radius of carrier pipe	2.25 in.
$r_2$ = outside radius of insulation	4.25 in.
$r_3$ = inside radius of carrier pipe	5.25 in.
$h$ = height of water in pipe	$0 < h < 10.5$ in.
$S$ = Concentration of leachable chlorides in the insulation (ppm by weight)	$50 < S < 200$

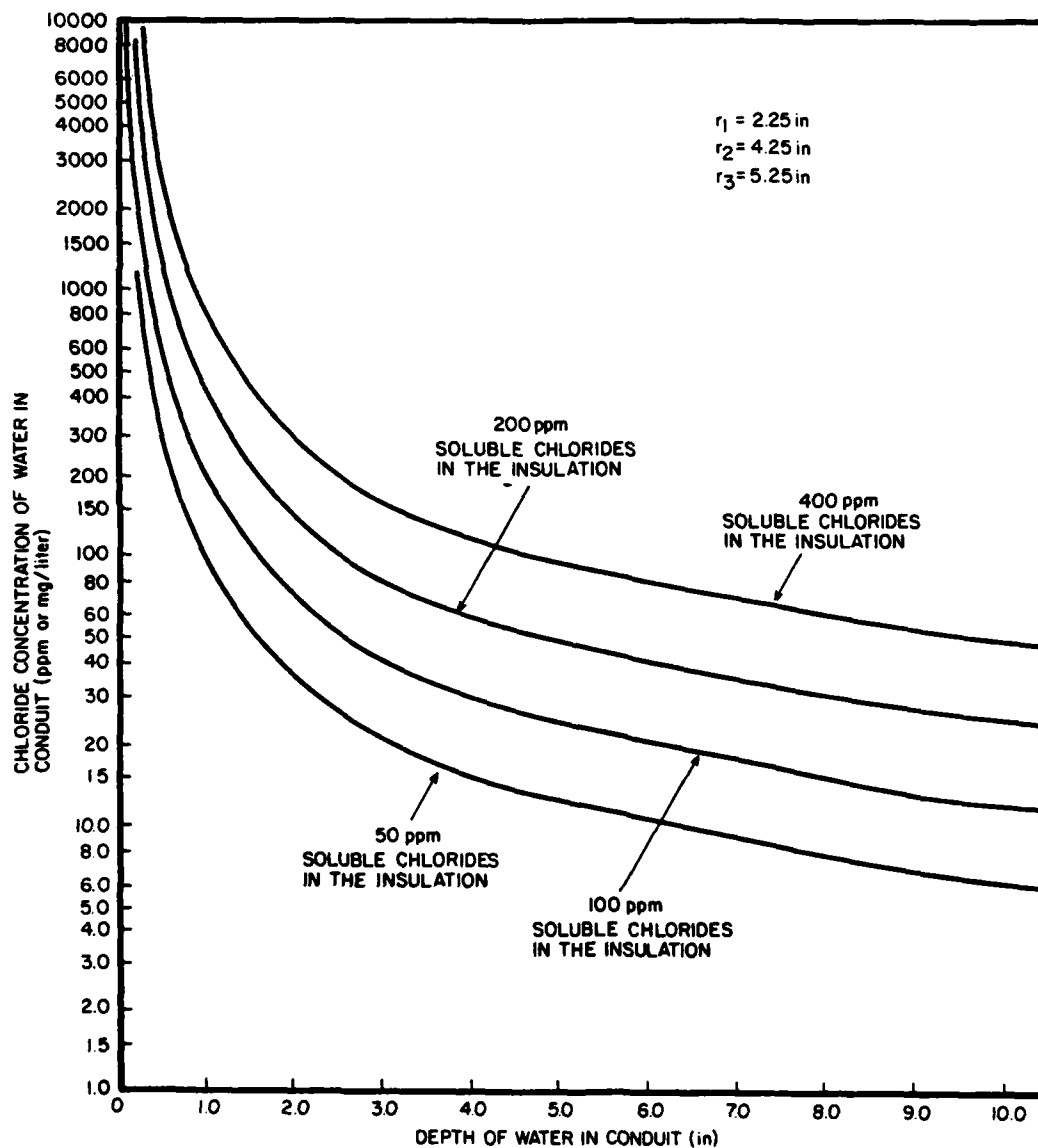
the conduit during installation, providing a positive seal against water entry into conduits from manholes, checking that conduit drain ports are properly sealed, providing adequate drainage for manholes, and requiring proper slope of the system to avoid low spots where water can collect. It may also be necessary to specify maximum allowable leachable chloride concentrations for insulation materials used in the system.

During routine operation and maintenance of underground heat distribution systems, corrosion problems can be alleviated by providing adequate manhole drainage, checking conduit drains periodically to see that they are closed, and checking conduit vents frequently to determine whether water has entered the system.

Figure 5 shows that once flooding of a conduit has occurred, promptly draining the system to flush out chlorides and other corrosive species is the most effective way to minimize damage to the system. During flooding an almost endless supply of aggressive ions can enter the system from the environment. The prompt repair of leaks is important not only to minimize the concentration of aggressive species in the conduit during boiling, but also to prevent the entry of additional corrosive species into the system from manholes, groundwater, or the heat-carrying medium.

## 5 CONCLUSIONS AND RECOMMENDATIONS

On the basis of this research, it is concluded that chlorides leaching from commercially available calcium silicate insulation can greatly contribute to the corrosion of underground heat distribution systems. This corrosion is aggravated if leaks in the system continue



**Figure 5.** Effect of changes in height of water in a typical conduit and in solubility of calcium silicate insulation on the concentration of chlorides flooding the conduit.

unchecked for long periods of time. Although minimal amounts of chlorides are released after 1 or 2 hours of boiling, the number increases dramatically after 7 or 8 hours, an amount of time which would be common if system flooding occurred.

To reduce the detrimental effects on underground heat distribution systems of corrosive ions from insulation and from manholes and leaks in the system, the following procedures are recommended:

1. Prevent water from entering the system through manholes by providing and maintaining effective seals at manhole entries.
2. Install positive drainage throughout the system to prevent water from accumulating at low spots.
3. Minimize contamination of the system by keeping soil and debris out of the conduit during installation.
4. Periodically check systems for high water in manholes, evidence of water in the conduit, and evidence of corrosion.
5. Promptly drain conduits if flooding occurs.
6. Promptly repair leaks in the conduit or carrier pipes to prevent further contamination of the system from ions in the environment.

Further investigation in three areas might also be helpful in reducing this type of corrosion:

1. Establishment of stricter requirements regarding the acceptability of insulation materials based on their concentration of leachable chlorides.
2. Determination of acceptable levels of leachable chloride concentrations in military heat distribution systems.
3. Investigation of effects of moisture absorption by calcium silicate insulation during a system shut-down, including possible corrosive effects on carrier piping of not only conduit systems, but also systems above ground, inside of buildings, and in trenches.

It is recommended that the feasibility and usefulness of having such requirements be investigated.

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